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ANALYSIS OF NONIONIC DETERGENTS IN RAW SURFACE WATERS BY THIN-LAYER CHROMATOGRAPHY AND INFRARED SPECTROSCOPY

Doris Smith, et al

Federal Water Pollution Control Administration
Cincinnati, Ohio

May 1967

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WATER POLLUTION SURVEILLANCE SYSTEM
APPLICATIONS AND DEVELOPMENT REPORT

27

ANALYSIS OF NONIONIC DETERGENTS IN RAW SURFACE WATERS BY THIN-LAYER CHROMATOGRAPHY AND INFRARED SPECTROSCOPY

Doris Smith
James J. Lichtenberg

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DIVISION OF POLLUTION SURVEILLANCE
CINCINNATI, OHIO

MAY 1967

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THE AUTHORS

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ANALYSIS OF NONIONIC DETERGENTS IN RAW SURFACE WATERS BY THIN-LAYER CHROMATOGRAPHY AND INFRARED SPECTROSCOPY

I. INTRODUCTION

As a part of the surveillance program of the Federal Water Pollution Control Administration, sampling stations are maintained to gather the information required to determine levels of pollution. One type of pollution is that caused by discarded surfactants. The specific class of surfactants of interest here are the nonionics. Nonionic surfactants presently constitute more than 20% of the total sales of surface active agents in the United States. The use of these compounds as wetting agents, detergents, emulsifiers and penetrants has increased over 30% since 1959 (1). Of this class of surfactants the alkyl phenol polyoxyethylenes are the most widely used.

While no widespread pollution problem involving nonionic surfactants has been recognized, an isolated problem has required our attention for approximately four years. In this instance, water treatment plant operators noted foaming on the river being used as a source, as well as in the treatment plant. The quantity of anionic detergents was too low to be the cause of the foaming problem in itself. It was noted that the water no longer foamed after passing through a Water Pollution Surveillance System carbon adsorption cartridge [1]. Thus, infrared analysis of an extract of a

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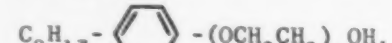
1. A description of the carbon adsorption cartridge is given on page 5.

carbon adsorption sample from the site was carried out. The spectrum suggested the presence of an alkylphenol polyoxyethylene type of compound. Comparison of a known alkylphenol polyoxyethylene and the sample analyzed by the phosphomolybdic acid (PMA) precipitation method of Oliver and Preston (2) coupled with infrared spectroscopy confirmed the presence of such a compound having the approximate structural formula shown below:



These compounds are commercially available as mixtures containing several ethylene oxide chain lengths. They are designated by the average length of the chain. Triton X-100 with an average chain length of 9-10 is commonly used. Studies by Huddleston (3) using the shake culture and river die-away tests have shown that branched chain and straight chain alkylphenol nonionics are relatively non-biodegradable; only the primary straight chain alcohol nonionics were found to be as biodegradable as the linear alkylate sulfonates (LAS).

During the investigation of the foaming problem, the need for a rapid and sensitive method for detection of nonionic detergents was recognized. The thin-layer method described below was developed to meet this need.

A number of gravimetric, volumetric and colorimetric methods are available for the determination of the ethylene oxide nonionic detergents in the higher concentration ranges. However, few methods are available for determination in quantities less than

1 ppm. In 1966, Burttschell (4) described a phosphotungstic acid (PTA) method with a sensitivity of 100 ppb in sewage. Also in 1966, Patterson et al. (5) described a thin-layer method with a sensitivity of 10 ppb for nonionic detergents in sewage. They report that as little as 1 to 5 μg of the nonionic detergent is visible on the thin layer using a modified Dragendorff spray reagent. This represented an improvement over similar methods of Mansfield and Locke (6) and Burger (7).

Ammonium cobaltothiocyanate has been widely used to form a complex with polyethoxylated-type compounds since it was first described by Gnamm (8). Van der Hoeve (9) (10), Wurzschmitt (11), Kurata (12), Brown and Hayes (13), Crabb and Persinger (14) and Greff, Setzkorn and Leslie (15) have all used this reagent in various compositions to measure the nonionics absorptiometrically. The present method uses this reagent to give the typical blue color reaction on the thin layer. The sensitivity and selectivity of the method are comparable to that of Patterson et al. The method is applied to carbon adsorption samples representing 1,000 to 20,000 liters of water as well as 1 liter water grab samples. Whenever possible, as with most carbon adsorption samples, the analysis is confirmed by the PMA or the PTA method and infrared spectroscopy. The procedures described below are currently in use at the Division of Pollution Surveillance laboratories in Cincinnati, Ohio.

II. EXPERIMENTAL

A. Reagents and Equipment

Unless otherwise specified, all reagents are Analytical Reagent grade.

1. Collection

Sample bottles and shipping containers, activated carbon cartridge and collection equipment - the details are given in Department of the Interior Publication WP-22 (16). Cliff Char 4 x 10 mesh carbon (Cliffs Dow Chemical Company, Marquette, Michigan) and Nuchar C-190 30 mesh carbon (West Virginia Pulp & Paper Company, New York) are used.

2. Extraction

Carbon tetrachloride - redistilled

Chloroform - redistilled

Acetone

300 ml Erlenmeyer flask, 5 dram vial, 50 ml beaker, 1500 ml beaker

2 liter separatory funnel for grab samples

large Soxhlet extractor for carbon adsorption samples (Corning Cat. No. 3885)

15 ml graduated centrifuge tubes

3. Thin-Layer Chromatography

Silica gel-G (Research Specialties)

Desiccator

Developing tank

Solvent System I - Benzene/cyclohexane/diethylamine (5:2:2)

Solvent System II - Butanone (MEK) saturated

with distilled water

Spray reagent - Ammonium cobaltothiocyanate- Dissolve 0.3 g of cobalt nitrate and 20 g of ammonium thiocyanate in 100 ml of distilled water

4. PMA Precipitation - Infrared Spectroscopy

Phosphomolybdic acid - Dissolve 10 g in 100 ml of distilled water

Barium chloride - Dissolve 10 g in 100 ml of distilled water

Hydrochloric acid (conc) diluted (1:4)

Filter paper - 11 cm, Grade E & D 15

250 ml beakers

Hot plate

Infrared spectrophotometer

5. Standard Nonionic Detergents - 1 mg/ml in chloroform

E.g., Triton series (Rohm & Haas Co., Philadelphia, Pa.) and Surfonic Series (Jefferson Chemical Co., Houston, Texas)

B. Procedure

1. Sample Collection

Carbon adsorption samples are collected by passing 1,000 to 20,000 liters of water through activated carbon at rates of 1/32 to 1/2 gpm. The carbon adsorption cartridge consists of a Pyrex glass pipe 3 inches in diameter and 18 inches in length. The cartridge is packed with all fine C-190 carbon (30 mesh) when using the low flow rate and low total throughput system. It is

packed with 4.5 inches of coarse carbon (4 x 10 mesh) on each end and 9 inches of C-190 carbon in the center when using the high flow rate and high total throughput system. The latter arrangement helps to prevent clogging when sampling turbid waters.

The details of the sampling equipment, its installation and operation are given in Department of the Interior Publication WP-22 (16).

Water grab samples are collected in 1-quart glass bottles equipped with screw caps fitted with teflon liners. Duplicate samples are collected and shipped to the laboratory in polystyrene cartons (16).

2. Extraction

Carbon adsorption samples are air dried in an oven at 40^{o} C for about two days. The carbon is then extracted with chloroform (approximately 2500 ml) in a large Soxhlet extractor for 35 hours. The extracting solvent is reduced to about 200 ml by distillation, filtered through solvent-washed filter paper into a 300 ml Erlenmeyer flask and evaporated to about 20 ml on a steam bath. It is then transferred to a tared 50 ml beaker and evaporated to dryness at room temperature and weighed.

The measured water grab samples are poured into a 2 liter separatory funnel and extracted three times with 75 ml aliquots of carbon tetrachloride. The aliquots are combined in a 250 ml beaker and evaporated to near dryness on a steam bath.

3. Thin-Layer Chromatography

Layers of silica gel 0.25 mm thick are prepared on 200 x 200 mm glass plates. A thin slurry of 30 g of silica gel G in 60 ml of water is spread over five plates with the aid of a variable thickness device. The plates are allowed to stand for five minutes, then dried in an oven at 100° C. for one hour. They are stored in a desiccator for future use. Layers stored longer than two weeks should be reactivated before use. Just prior to use, marks are made near the edge of each plate at distances of 1.5 cm and 11.5 or 16.5 cm above the bottom edge to define the spotting line and the point at which the solvent front has moved 10 or 15 cm depending on the solvent system used. A line is etched across the silica gel layer at the 11.5 or 16.5 cm mark.

Ideally, two solvent systems are employed for

development. In the case of 1 liter samples, this usually requires the extraction be carried out in duplicate. With solvent system I, benzene/cyclohexane/diethylamine (5:2:2), the nonionic detergent mixture moves as a single spot and is matched with standards for quantitative determination. With solvent system II, water-saturated MEK, the nonionic mixture separates into its components producing a "polyoxyethylene spectrum".

The developing solvents are added to the tanks which are lined with filter paper wicks and allowed to equilibrate for one hour before use. Solvent system I is replaced after every second development and again allowed to equilibrate. The developing time for solvent system I is about 18 minutes. For system II it is about 40 minutes.

The gross CCE, or its water soluble fraction, obtained by a Shriner and Fuson solubility class separation (17) is analyzed. More of the sample can be spotted and, thus, greater sensitivity achieved when the water soluble fraction is analyzed. Details of the specific solubility separation are given in Department of the Interior Publication WP-22 (16).

The water soluble fraction is dissolved in

chloroform, transferred to a 15 ml graduated centrifuge tube and evaporated to an appropriate volume for spotting, usually 1.0 ml. As much of the sample as possible is spotted, taking care not to overload the thin layer. A gentle stream of air is directed on the point of application to keep the diameter of the spot to a minimum. The diameter of the spot should not exceed 1.0 cm. Up to 20-30% of the water soluble fraction can be spotted. Varying concentrations of standard nonionic detergents are also spotted for matching with the sample. Triton X-45, Triton X-100, Triton X-102, Surfonic N-150 and Surfonic N-300 are commonly used standards in this laboratory. Two silica gel layers are spotted. One is placed into the tank containing solvent system I and developed to the 10 cm line. The other is placed in the tank containing solvent system II and developed to the 15 cm line.

The plates are then removed from the tank and allowed to dry thoroughly at room temperature. The thin layer is carefully sprayed with a light even coat of aqueous ammonium cobaltothiocyanate reagent. The layer may be resprayed, if necessary, after allowing the first application to dry. However, over-spraying may cause the spots to spread and a large excess will cause the silica gel layer to

run.

The nonionics appear as bright blue spots almost instantly, and the color intensity reaches a maximum when the silica gel layer is just dry. Quantitative estimation is made at this time since the spots gradually fade and the background darkens obscuring the nonionic spots. At higher concentrations the spots remain distinct for several hours. Covering the layer with a clean glass plate will retard the fading. Quantitative estimation is made by visually comparing the intensity of the sample spots to that of the standards.

The extract of the water grab sample in carbon tetrachloride is transferred to a 15 ml graduated centrifuge tube and reduced to an appropriate volume for spotting, usually 0.1 ml. The entire sample is spotted whenever possible. This can usually be accomplished with the extract of a 1 liter sample. However, care must be taken not to overload the silica gel layer. Duplicate samples are spotted and developed in systems I and II as described above.

4. Infrared Determination

Infrared analysis of the phosphomolybdic acid complex described by Oliver and Preston (2) is used for qualitative confirmation of the thin

layer results. There is usually not sufficient compound available in a 1 liter surface water sample for this determination. The infrared method is best suited to analysis of the water-soluble fraction of the CCE. The gross extract may be used but gives less satisfactory results.

The water-soluble fraction is dissolved in chloroform and a measured aliquot (approx. 1/2, 1/3 of a CCE) is transferred to a 250-ml beaker and evaporated to dryness. To this is added 5 ml of HCl, 5 ml of $BaCl_2$ solution and 5 ml of phosphomolybdic acid solution. The solution is diluted to 150 ml with distilled water and brought to a boil on a hot plate. The complex forms a voluminous yellow-green precipitate. The beaker is covered with a watch glass and allowed to stand overnight (18 hours). The time may be varied, but the treatment should be uniform for all samples and standards. The precipitate is filtered through a Buchner funnel, washed with distilled water and dried in a desiccator. Qualitative infrared spectra of the complex are obtained in a mineral oil mull or in a KBr pellet.

III. RESULTS AND DISCUSSION

Developing Solvent

Varying combinations of several solvent systems were studied. As noted by Patterson et al. (5), the nonionic substances tend to remain at the origin when single solvents are used. An exception to this is diethylamine which moved all of the nonionics studied with the solvent front. The proper solvent mixture provides good resolution of the nonionics. Solvent system I, benzene/cyclohexane/diethylamine (5:2:2) was found to be the most satisfactory for comparison of samples with standards for quantitative determination. With this system, the nonionic mixture appears as a single compact spot. The sample spots do not usually appear as well defined as those of the standards. Some tailing and spreading occurs, particularly with CCE samples. Figure 1 shows a chromatogram of several standard surfactants of the alkylphenoxy polyethoxy ethanol series developed with solvent system I. It is seen that the length of the oxyethylene chain does not significantly influence the movement up the thin layer. However, it was noted that Hodag E-30 (30 ethylene oxide groups) tends to streak. Figure 2 shows CCE water soluble fractions and X-100 standard nonionic from 10 to 100 μg quantities developed in solvent system I. Sample 1 was spotted in 30, 40, and 60 μg quantities

of a total of 300 µl of sample. All organics, which are visible, remained at the origin. Sample 2 shows the effect of overloading. It was spotted in 10, 30, and 50 µl volumes of a 1000 µl sample. It is much cleaner than sample 1 but contains a considerably larger quantity of nonionics. In this case the sample should be respotted in smaller quantities for quantitative determination. The residual spots at the origin may be due to high molecular weight polyethelene glycols, quaternary amines or alkylbenzenesulfonates which react with the reagent but do not move in this system. Due to evaporation losses, the composition of solvent system I changes rather rapidly resulting in progressively poorer chromatograms. For best results the solvent should be changed after every second run. When a large number of samples are to be run several developing tanks are prepared in advance. Solvent system II, water saturated methyl ethyl ketone described by Burger (7), provided excellent separation of the nonionic components as shown in Figure 3. Standard nonionics with varying ethylene oxide chain lengths were spotted. The compounds with shorter chain lengths move up the layer further and the individual components separate more widely than the longer chain lengths. E-30 which contains 30 ethylene oxide groups remained at the origin. This system is used to confirm

the nonionic identification. In addition, the average oxyethylene chain length can be estimated as suggested by Burger (7). Figure 4 shows CCE samples and standards X-100 and N-150 developed in solvent system II. Samples 1 and 3 separated into the typical polyoxyethylene spectrum. Too large a quantity of sample 2 was spotted and no separation is noted. It appears to be a compound similar to N-150, perhaps with slightly longer chain length but in much higher concentration than the standard. Figure 5 shows three different carbon samples also run in solvent system II along with standard X-100 and X-45. The three samples separated into the typical spectrum. The yellow background in the carbon extracts may be a source of interference in solvent system II. However, the blue areas at the top of the chromatogram appear 10 to 15 minutes after spraying and are thus distinguished from the nonionics which appear almost instantly.

Several trials using the ethyl acetate/water/glacial acetic acid (4:3:3) mixture of Patterson et al. were not as satisfactory as the solvent system selected. The system of Patterson cannot be used with the ammonium cobaltothiocyanate spray reagent since the silica gel in the area of the spots turns brown-red making it difficult to recognize the nonionic detergent spots.

Spray Reagent

Table 1 lists reagent compositions that have been used to measure nonionic compounds absorptiometrically. The spray reagent composition found to be most satisfactory for thin-layer chromatography was 200 g of ammonium thiocyanate and 3 g of cobalt nitrate hexahydrate per liter. More concentrated reagent solutions produce a dark blue background on the silica gel layer which masks the non-ionic compounds. The nonionics appear as bright blue spots almost instantly, and the color intensity reaches a maximum when the silica gel layer is just dry. Quantitative estimation is made at this time. The spots gradually fade after this, but quantities of 20 μg remain visible for several hours.

The modified Dragendorff reagent of Patterson et al. worked well with water grab samples but showed less contrast than ammonium cobalthothiocyanate when used with CCE samples. Thus, the latter reagent is preferred. It was found that layers sprayed with the Patterson reagent could be over-sprayed with ammonium cobaltthiocyanate after the first reagent had faded. This did not affect the results. Over-spraying the ammonium cobaltthiocyanate with Patterson's reagent produces a dark brown color which obliterates the nonionic compounds.

Interferences

The reagent is not specific for the polyoxyethylenes. However, compounds such as polyethylene glycols, quaternary

amines and alkylbenzenesulfonates which react with the reagent remain at the origin and do not interfere with the thin-layer determination. Other chemicals which might be expected to interfere were also tested. They generally move with the solvent front, remain at or near the origin or give an improper color reaction. Still others, which occur mainly in the CCE samples produce a belated reaction approximately 10 to 30 minutes after spraying and are thus distinguished from the nonionics which produce bright blue spots almost instantly. Thus, ammonium cobaltothiocyanate is sufficiently selective for the determination of this class of compounds.

The possibility of interference by naturally occurring substances was also considered. Table 3 lists several natural substances that were extracted and tested on the thin layer. As noted in the table, several natural materials produced a positive reaction with ammonium cobaltothiocyanate, but they do not occur in the same area of the chromatogram and in nearly all cases they appear 10-15 minutes after spraying. Hence, they do not interfere with the analysis of the polyoxyethylene compounds tested.

Extraction Efficiency

The efficiency of adsorption on and desorption from carbon for the nonionic compounds is unknown. Thus, the carbon adsorption method (CAM) is not considered

quantitative. All data obtained by this method are minimum values, the actual values being equal to or greater than the reported values.

Quantitative recovery of nonionics from water grab samples requires that they be salted-out. Saturation of the samples with sodium chloride is suitable for this purpose (4)(5)(15). About 25 g of NaCl are required when extracting a 100 ml sample. Patterson et al. (5) use 150 g of magnesium sulfate when extracting a 250 ml sample. Greff et al. (15) studied the effect of sample volume on extraction efficiency. They found that as the volume of the salt saturated sample increased the extraction efficiency decreased markedly. In light of this and because of the large quantity of salt required for extraction of a 1 liter sample we have not included this operation in our limited work on water grab samples. The recoveries of nonionics omitting this step range from about 65% for readily water soluble Triton X-100 to 90% for poorly water soluble Triton X-45. These recoveries were obtained from distilled water samples dosed with 30 to 80 μg/l of standard nonionic compounds. An alternate approach, that of evaporating the 1 liter sample to 100 ml and applying the salting-out technique may provide more quantitative recoveries. We are presently examining this possibility.

Sensitivity

The sensitivity of the thin layer method varies somewhat with the average oxyethylene chain length. As the chain length increases, the sensitivity increases. This is shown in Figure 6 where varying concentrations of nonionic detergents of the Triton and Surfonic series are chromatographed. The lower limits of detection for the Triton and Surfonic series on the thin layer are listed in Table 2. It is seen that the limit for the most commonly used nonionics (9-10 oxyethylene groups) is about 5 μg. The Triton series discussed here are all octylphenoxypolyethoxy ethanols, and the Surfonic series are all nonylphenoxypolyethoxy ethanols.

Considering the lower limit of detectability on the thin layer as 5 μg and the volume of water sampled as 1000 liters, the sensitivity of the CAM method is considerably less than 0.1 μg/l. The sensitivity for 1 liter grab sample is then in the neighborhood of 5 to 10 μg/l.

Infrared Spectroscopy

Infrared spectroscopy is used to substantiate the identification of the nonionic compounds. Figure 7 shows the spectra of a CCE water soluble fraction and standard Triton X-100. The characteristic ether band is noted at 1105 cm^{-1}. Several other bands common to both sample and standard are also noted. Figure 8 shows

spectra of the PMA complex of the same sample and standard. The typical strong PMA bands at 807, 880, 960 and 1065 cm^{-1} as well as the other bands present are common to both sample and standard.

In a mineral oil mull, approximately 300 μg of Triton X-100 is required to produce a satisfactory spectrum using a Perkin-Elmer Model 137B Infracord. Using a micro KBr pellet and X6 scale expansion and a Perkin-Elmer Model 421 infrared spectrophotometer, as described by Burttschell (4), notable less than 300 μg of the compound is required.

IV. SUMMARY

A thin layer chromatographic method using ammonium cobaltothiocyanate for detection of low ppb quantities of nonionic surface active agents has been presented. The method has been applied to surface water samples obtained by the carbon adsorption and grab methods. Two solvent systems are employed for the TLC development. Solvent System I is used to quantitate the nonionic surfactant by comparing the intensity of the sample spots to that of standards. Solvent system II is used to develop the typical "polyoxyethylene spectrum" and thus confirm the presence of the nonionic compounds. When sufficient sample is available, a PMA-IR technique is used for additional verification of the results.

A survey for nonionic surfactants in the Nation's major interstate waterways is now underway using the method described. Carbon adsorption samples from approximately 130 Water Pollution Surveillance System stations are being analyzed. Historical, i.e., samples collected and stored since 1957, as well as current samples will be included in the survey.

Acknowledgement

The authors wish to acknowledge the assistance of Dr. A. A. Rosen and R. H. Burttschell of the Robert A. Taft Sanitary Engineering Center in making the initial identifications of nonionic detergents in the CAM samples and Dr. A. W. Breidenbach for reviewing the manuscript. Dr. C. I. Weber of our laboratory photographed the thin layer chromatograms. Samples of commercial nonionic detergents and technical literature were supplied by the Rohm and Haas Co., Philadelphia, Pa., the Jefferson Company, Houston, Texas and the Hodag Chemical Company, Skokie, Illinois.

Mention of products and manufacturers is for identification only and does not imply endorsement by the Federal Water Pollution Control Administration or the U. S Department of the Interior.

TABLE 1

Reported Reagent Compositions

Author	Ammonium Thiocyanate g/liter	Cobalt Nitrate Hexahydrate g/liter
Gnamm (8)	347	1.4
Van der Hoeve (9) (10)	174	2.8
Wurzschmitt (11)	174	28
Brown & Hayes (13)	200	30
Greff et al. (15)	620	280
Kurata (12)	620	280
This Paper	200	3

TABLE 2

Lower Limit of Detection on the Silica Gel Layer

Compound	Average Chain Length	Detectable Limit (μg)
Triton X-45	5	10
Triton X-114	7-8	5
Triton X-100	9-10	5
Triton X-102	12-13	<5
Surfonic N-40	4	10
Surfonic N-60	6	10
Surfonic N-95	9.5	5
Surfonic N-150	15	<5
Surfonic N-300	30	<5

TABLE 3

Naturally Occurring Substances Extracted and Chromatographed on Silica Gel Thin Layers

Substance	Reaction With Ammonium Cobaltothiocyanate
Humic Acid	-
Abietic Acid	-
α and β-Conidendrin	-
Vanillin	-
Soil	+
Straw	+
Weed Blooms	+
Ragweed	+
Tree Leaves	+

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FIGURE 1

STANDARD NONIONIC DETERGENTS DEVELOPED

IN SOLVENT SYSTEM 1

(50 µg each)

Triton			Surfonic			Hodag	
X-45	X-100	X-102	N-40	N-95	N-150	E-8	E-30

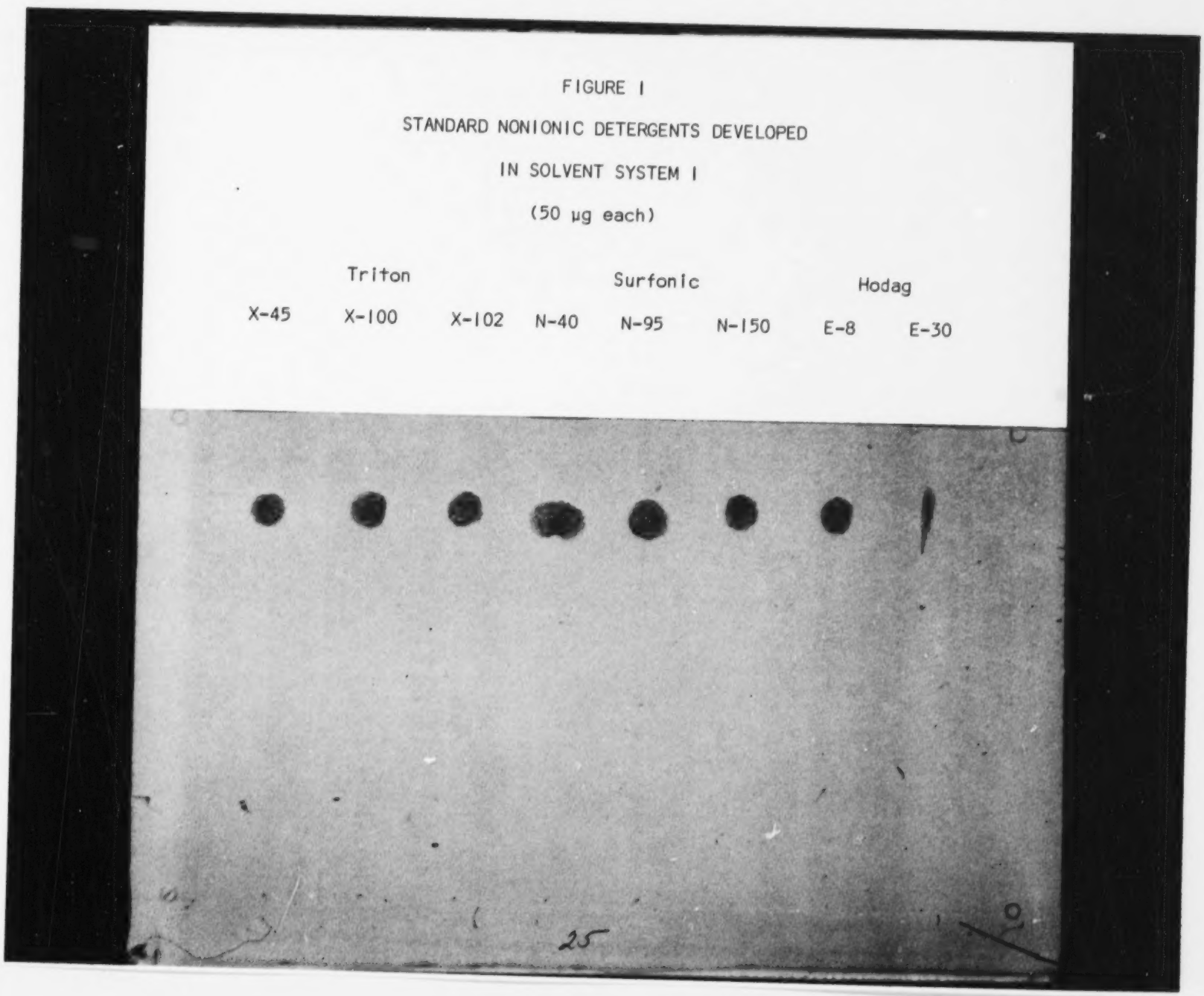

FIGURE 2

CCE WATER SOLUBLES DEVELOPED

IN SOLVENT SYSTEM I

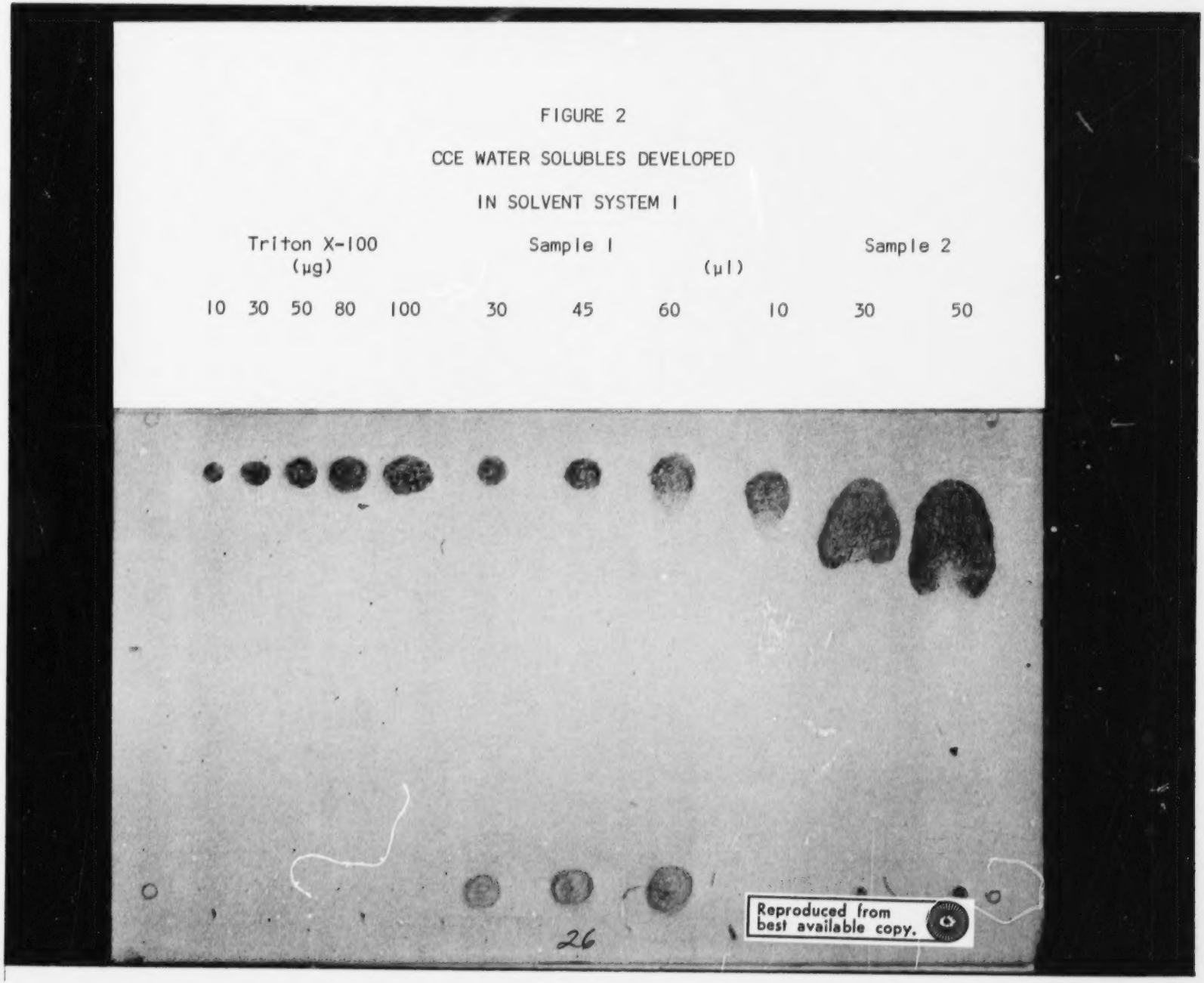

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FIGURE 3

STANDARD NONIONIC DETERGENTS DEVELOPED

IN SOLVENT SYSTEM II

(150 µg each)

X-45 X-100 X-102 N-40 N-95 N-150 E-8 E-30

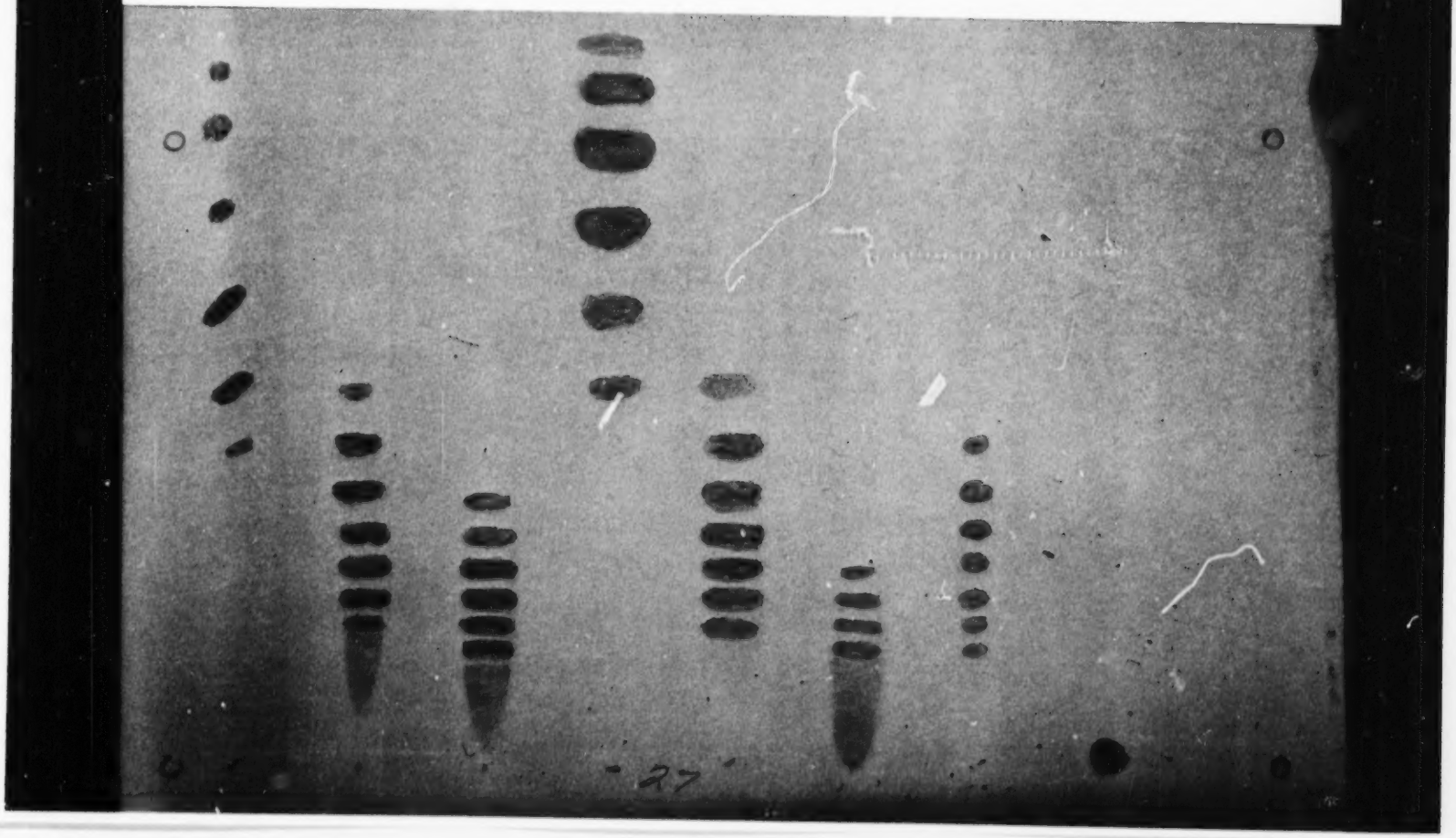


FIGURE 4

CCE WATER SOLUBLES DEVELOPED

IN SOLVENT SYSTEM II

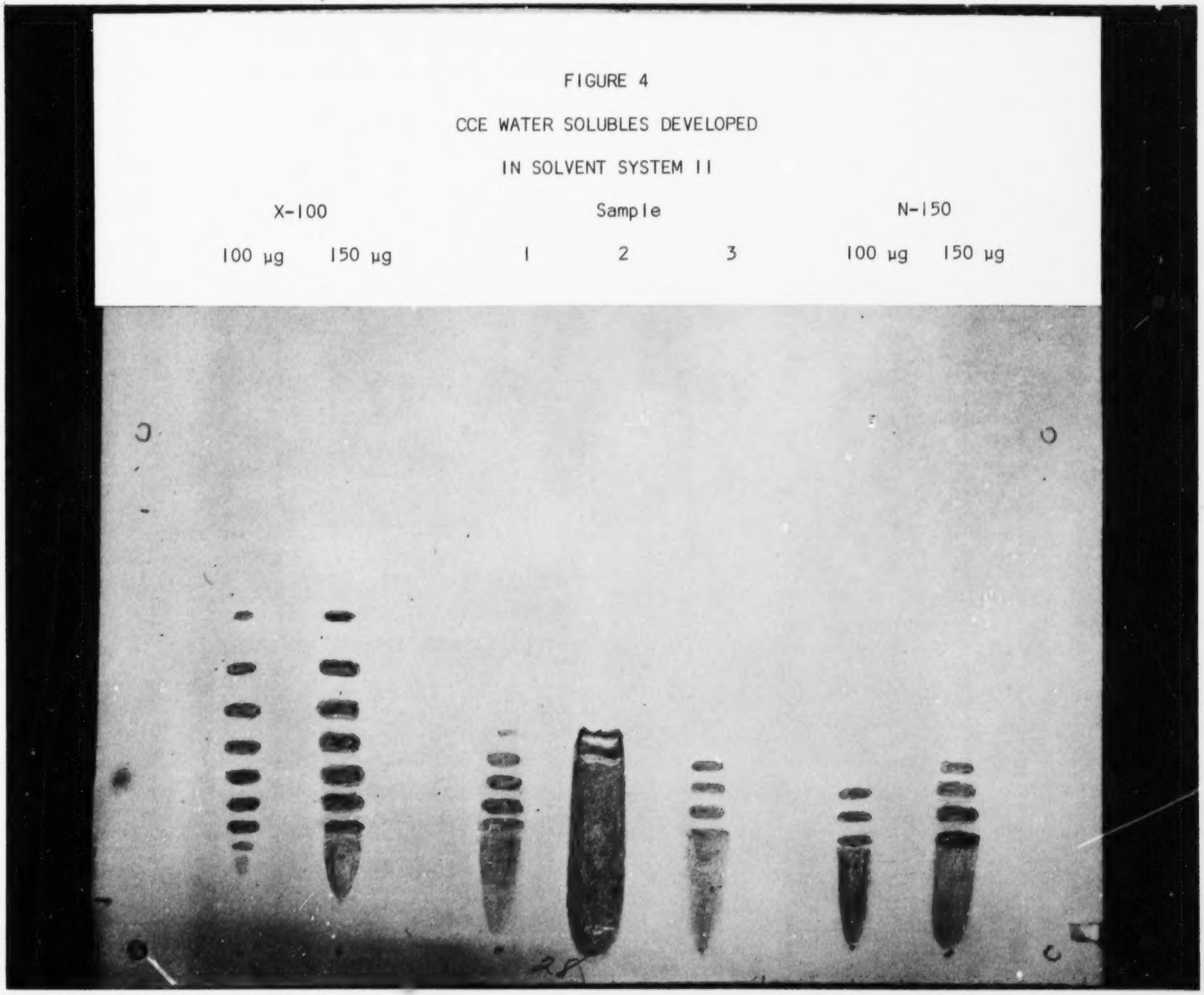


FIGURE 5

CCE WATER SOLUBLES DEVELOPED

IN SOLVENT SYSTEM II

FIGURE 6

STANDARD NONIONIC DETERGENTS DEVELOPED

IN SOLVENT SYSTEM I

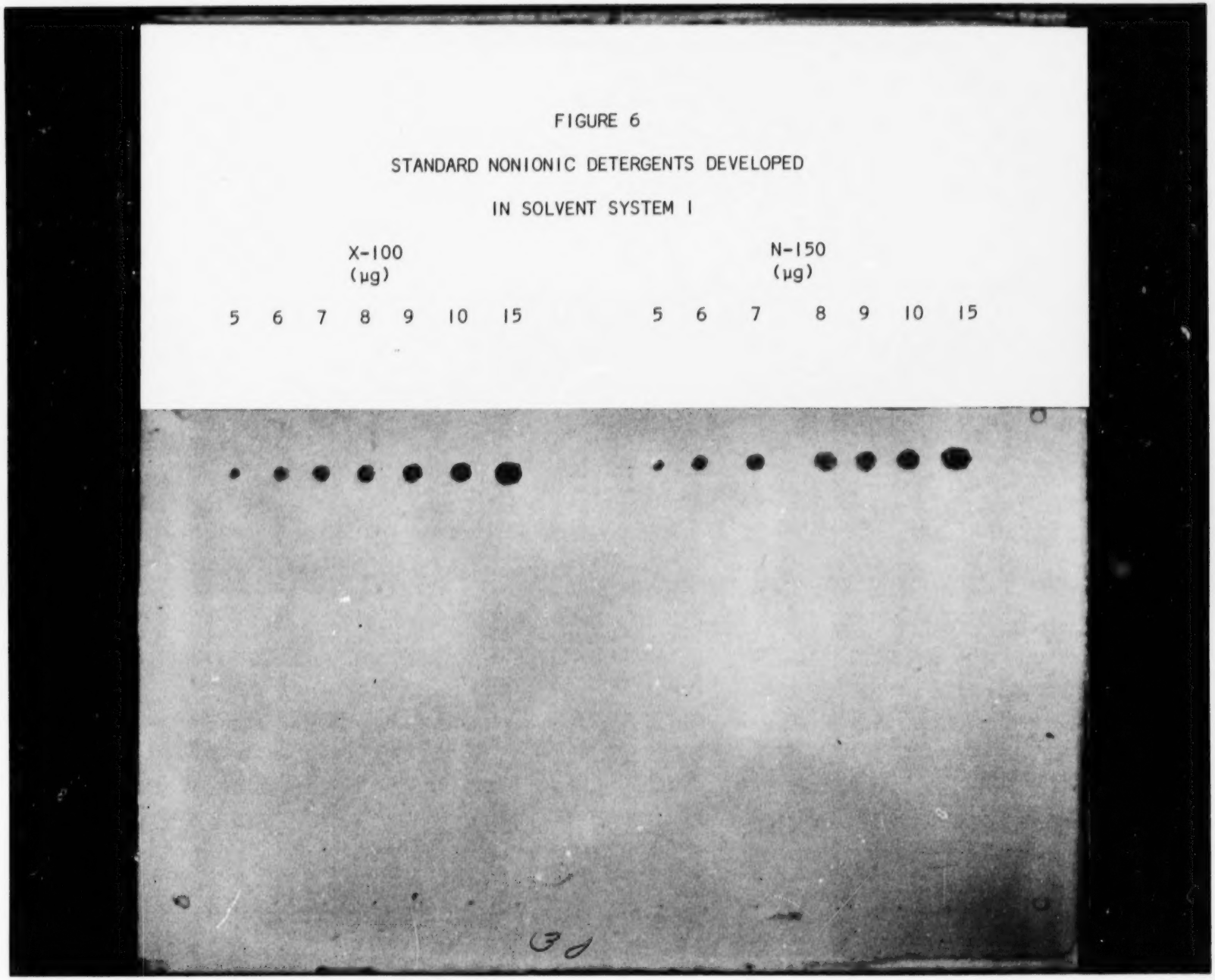
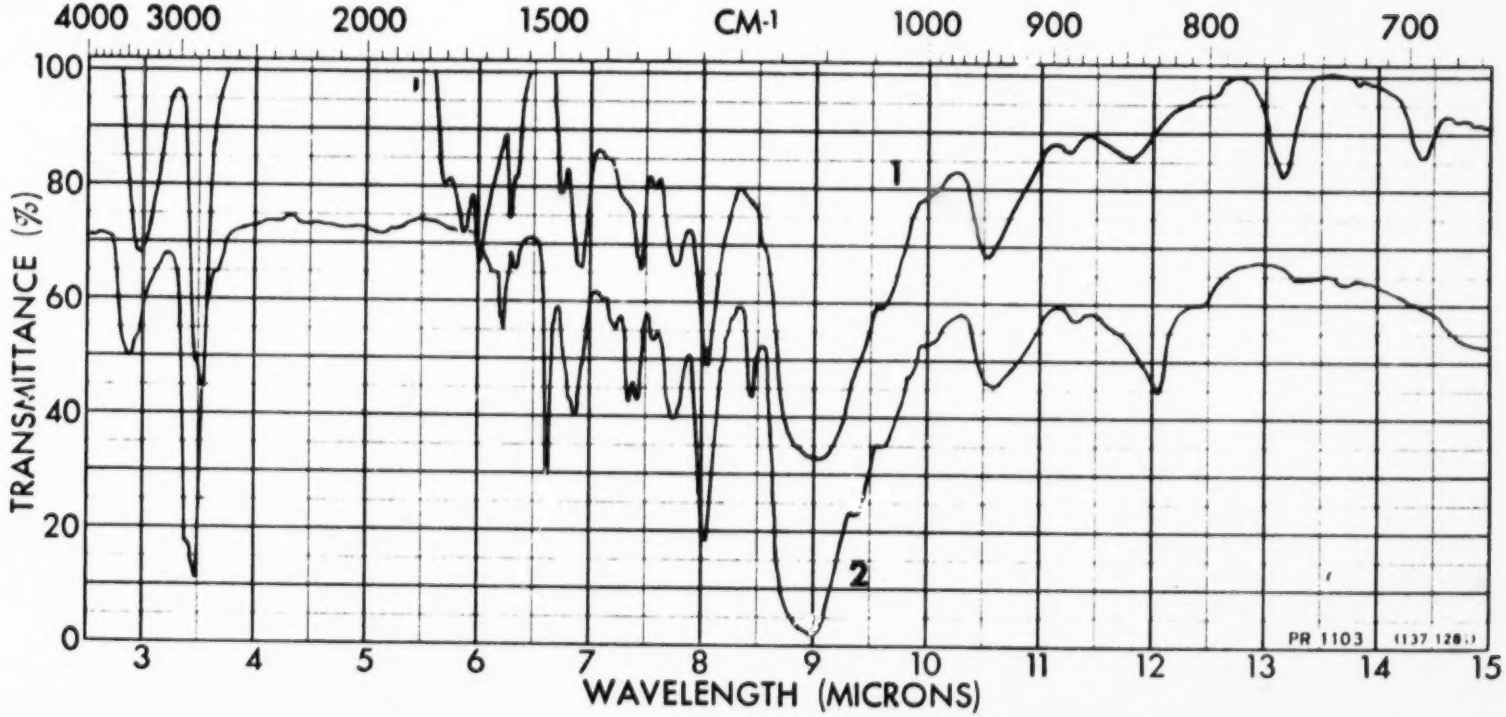


Figure 7

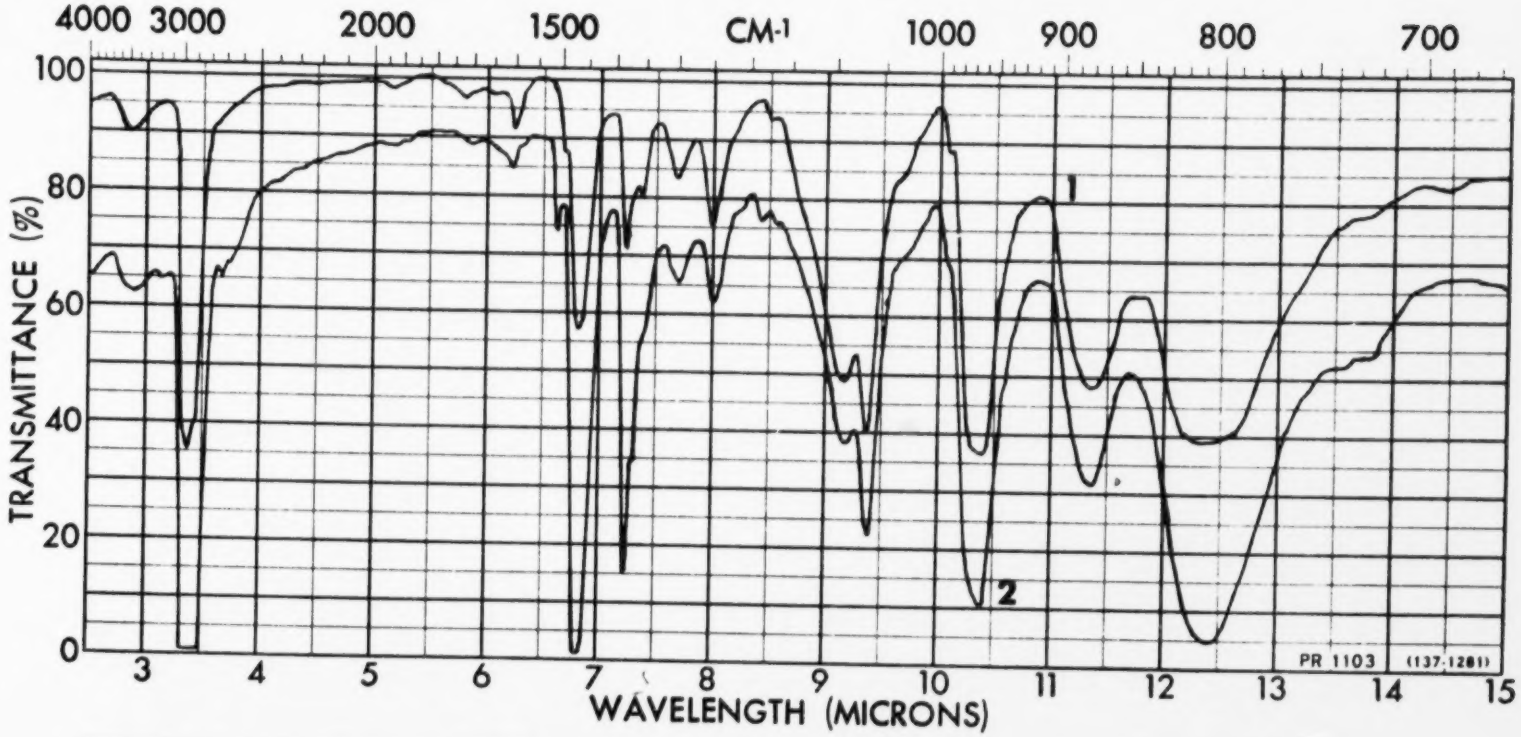


SPECTRUM NO.____	ORIGIN____	LEGEND____	REMARKS____
SAMPLE____	____	1. CCE Sample	Water Solubles
Normal State	PURITY____	2. Standard	Triton X-100
____	PHASE Sandwich	DATE____	____
____	THICKNESS____	OPERATOR____	____

SPECTRUM NO.____
SAMPLE____

RECORDING CHARTS

Figure 8



SPECTRUM NO.____	ORIGIN____	LEGEND____	REMARKS____
SAMPLE____	____	1. CCE Sample	Water Solubles
PMA Complex	PURITY____	2. Standard	Triton X-100
____	PHASE Mull	DATE____	____
____	THICKNESS____	OPERATOR____	____

SPECTRUM NO.____ SAMPLE____

RECORDING CHARTS
GRAPHIC CONTROLS CORPORATION
BUFFALO NEW YORK